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## Formulation and Evaluation of herbal shampoo using neem, amla and reetha extract

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**Abstract**

**Background:** The formulated shampoo contains amla fruit, which is a great source of vitamin A and various minerals which helps in reducing hair loss, neem leaf, helps in sealing hair follicles, treats head lice and conditions scalp and reetha fruit, an anti-inflammatory herb which is used to treat dandruff and other infection of the scalp.

**Aim:** The aim of this study is to evaluate and formulate herbal shampoo containing all natural components with an emphasis on efficacy and safety.

**Methods:** Leaves of *Azadirachta indica* and fruits of *Phyllanthus emblica* and *Sapindus mukorossi* were used in the formulation and subjected to Physical properties and appearance pH determination, wetting test, skin irritability, forms formation and stability.

**Conclusion:** The evaluation parameters were fulfilled and shown in acceptance range and can be further instigated for production and testing.

**Result:** The prepared herbal cleanser was found to be in the stipulated pH range, good stability, cleansing property, foam formation, viscosity, density, no skin irritation and microbial growth.

**Keywords:** Herbal Shampoo, *Azadirachta indica*, *Phyllanthus emblica*, *Sapindus mukorossi*, evaluation

**Introduction**

Hair is an important part of overall appeal of human body. There are many hair problems like thinning of hair, lack of hair volume, immature graying, conditioning, hair loss etc. have been observed by most of the individuals. Shampoo can also be defined as a cosmetic preparation used for washing scalp and hair packed in a form which is convenient for use [1].

**Hair structure**

Hair, protective appendages on the body and the structure of integument with sebaceous glands, sweat glands and nails are considered an important part of a body, derived from the skin ectoderm. They are also known as epidermal derivatives, since they originate from the epidermis during embryological development [2].

**Hair fall:** It is thinning of hair on scalp. Alopecia is the term for hair fall, it can be permanent or temporary. Hair fall is the most common problem among all patients. Hair fall can occur due to various problems such as poor diet i.e., less protein intake etc., excessive hair colouring and styling, physical stress, deficiency of vitamin B and taking excessive amount of vitamin A. To overcome this problem many hair growth promoters can be used such as *emblica officinalis* (amla), *ocimum sanctum* (tulsi) etc [3].

Herbal shampoo is a widely used daily unstable product all over the world. Herbal shampoos are defined as a preparation of surface-active material (surfactant) in suitable form solid, powder, or liquid which when used under the conditions specified will remove dirt, grease from the scalp and hair. It contains all natural ingredients with herb extract. It helps to improve the quality of hair by providing shine, moisture, growth and strength to hair roots [4].

**Materials and Methods****Plant collection**

The leaves of *Azadirachta indica* were collected from Siliguri, West Bengal. After that the leaves were shade dried and coarsely powdered using mortar and pestle for extraction using maceration technique.

The fruits of *Phyllanthus emblica* and *Sapindus mukorossi* were brought from the local market of Siliguri, West Bengal. After that the fruits were shade dried and coarsely powdered using mortar and pestle.

**Chemicals required**

Chloroform, gelatine, 0.1M sodium chloride, guar gum, glycerine, vitamin E capsule, rose oil, lemon oil, activated charcoal, petroleum ether, distilled water.

**Preparation of solvent extract**

- **Extraction of *Azadirachta indica***

- a. Fresh neem leaves are collected and shed dried for 15 days.
- b. The dried leaves are then powdered using a motor and pestle.



**Fig 1:** Filtration process of Neem

- c. The Powdered Neem leaves are sieved and weighed 24.45 g and macerated in a beaker using 200 ml of distilled water with continuous stirring.
- d. The prepared mixture is kept covered with aluminium foil and kept for 3 days for maceration while stirring in between, and then the mixture was filtered using a filter paper.
- e. The excess solvent is evaporated using a Rotary evaporator and then the remaining mixture was dried on a hot water bath.
- f. The dried extract was collected and kept in desiccator for cooling.
- g. The prepared extract is weighed.



**Fig 2:** Maceration of Neem

- **Extraction of *Phyllanthus emblica***

- a. Fresh amla fruit were finely chopped and shed dried for 15 days and powdered using mortar and pestle.
- b. The powered amla weigh 17.02g mixed with 0.25ml chloroform in 100ml water.
- c. Stir it for 15mins and macerate it for 3 days while stirring in between.
- d. Filter the solution and allow it to evaporate in rotary evaporator.
- e. Take the filtrate out of rotary evaporator and completely dry it on hot water bath.
- f. Keep the extract in desiccator for cooling.
- g. Collect and weigh the obtained extract.

- **Extraction of *Sapindus mukorossi***

- a. Fresh reetha fruit is collected; seed is removed and chopped finely using a clean knife.
- b. The chopped fruit is shed dried for 3 weeks.
- c. The dried fruit is then powdered using a mixer grinder; the prepared powder is sieved so as to remove any large pieces of the fruit.
- d. The fine powder is then weighed 17.15 gm.
- e. Measured 100 ml of petroleum ether using a measuring cylinder, transfer it to a beaker and add the prepared powder.
- f. Stir the mixture and cover it with an aluminium foil and macerate it for 3 days.

- g. After that the macerated mixture is filtered using a filter paper and the filtrate is then kept on hot water bath for drying.
- h. The completely dried extract is then collected and kept in desiccator for cooling.
- i. The cool extract is then weighed on a digital weighing machine.



**Fig 3:** Separation process of neem extract using Rotary Evaporator

**Formulation of Herbal Shampoo:****Procedure**

1. Take 3.5 ml of 0.1M sodium chloride in a beaker.
2. Add 1gm of guar gum to the beaker
3. Weigh 0.1ml of glycerine and add it to the beaker

4. Add 3.5ml gelatine, one capsule of vitamin E and 1gm of activated charcoal powder to the beaker.
5. Add 2.55 gm of reetha extract, 5gm of amla extract and 1.95 gm of neem extract and mix it well
6. Add water as required to make it a smooth and uniform paste.
7. Now add 2-4 drops of rose oil to the mixture.
8. Add 1drop of lemon oil as a preservative.
9. Continue to stir it for some times to avoid formation of any lumps
10. Allow it to cool and evaluate.

### Evaluation of herbal shampoo

To evaluate the prepared formulation, quality control test including visual assessment and physico-chemical controls such as P<sup>H</sup>, density, viscosity, surface tension, foam volume, foam stability and wetting time was performed using standard protocol.

1. **Physical appearance/visual inspection:** The formulation prepared was evaluated for the clarity, colour, odour and foam producing ability and fluidity.
2. **Determination of P<sup>H</sup>:** A 10% v/v shampoo solution was constituted in distilled water and the P<sup>H</sup> of the solution was measured by using a calibrated P<sup>H</sup> meter
3. **Determination of solid content percentage:** A clean dry evaporating disc was weighed and 4gm of shampoo was added to the evaporating disc. The evaporating disc with shampoo was placed on the hot plate until the liquid portion was evaporated. The weight of the solid content present in the shampoo was calculated after drying.
4. **Wetting time:** Wetting time was calculated by noting the time required by the canvas paper to sink completely. A canvas paper weighing 0.42gm was cut into a disc of diameter measuring 1 inch. Over the shampoo [1%v/v] surface, the canvas paper disc was kept and the time taken for the paper to sink was measured using the stopwatch.
5. **Cleansing action:** The cleansing property of the herbal shampoo was evaluated by the application of the shampoo on hair that has not been washed for 7 days. The shampoo was used to wash the hair of human subject that had applied oil 4-5 hours before washing. The performance of the shampoo was assessed on its ability to remove oily dirt from scalp.
6. **Foaming ability and foam stability:** Cylinder shake method was used for determining foaming ability. 50ml of the 1% herbal shampoo solution was put into a 250ml graduated cylinder and the cylinder was covered with hands and shaken for 10 minutes. The total volume of the foam content after 1min shaking was recorded. Immediately after shaking the volume of the foam at 1min intervals for 10 minutes were recorded. The foam volume remains same throughout the period of about 5min showing that the generated foam by the shampoo has good stability and the prepared shampoo exhibits higher foam property which may be due to the presence of soapnut. 1ml shampoo is dissolved with 2ml water and shaken vigorously for 10 minutes produced 0.4ml foam.
7. **Stability study:** The stability of the formulation was studied for a period of 4 weeks by keeping at temperature of 25-30°C.
8. **Skin irritation test:** Prepared herbal shampoo was applied on skin for 5 minutes after that was washed and tested for irritation or inflammation to the skin.

9. **Conditioning attributes:** The conditioning effect of the shampoo on hair was evaluated after the hair had been washed with it. Conditioning properties include all desirable benefits imparted to the hair such as increase mass to the hair, improved lusture, softness and silkiness.
10. **Viscosity:** Viscosity of shampoo was determined by using Ostwald's viscometer. The viscosity of herbal shampoo was measured by counting drops of herbal shampoo from the mark to bottom.

$$\text{Viscosity} = \frac{\text{Density of shampoo} \times \text{Timing of runoff of shampoo}}{\text{Density of water} \times \text{Timing of runoff of water}} \times \text{viscosity of water}$$

11. **Density:** First take empty weight of pycnometer, then fill it till neck with shampoo and then weigh it along with shampoo. Again, fill the pycnometer with water and weigh it.

$$\text{Density} = \frac{\text{Weight of pycnometer with shampoo} - \text{Weight of empty pycnometer}}{\text{Weight of pycnometer with water} - \text{Weight of empty pycnometer}}$$

12. **Microbial examination:** 1ml of shampoo was poured to sterile petri dish under aseptic condition and then allowed to set. The plates were incubated at 37°C for 24 hours and observed for microbial growth.

Evaluation parameters of herbal shampoo	
Organoleptic evaluation	Colour Odour Taste Texture Conditioning performance
Physicochemical evaluation	Ash value Total ash Acid insoluble ash Moisture content P <sup>H</sup>
Cleaning action Foaming index Wetting time Solubility Dirt dispersion Stability study	
Safety parameters	Eye irritation test Skin sensitization test

### Result

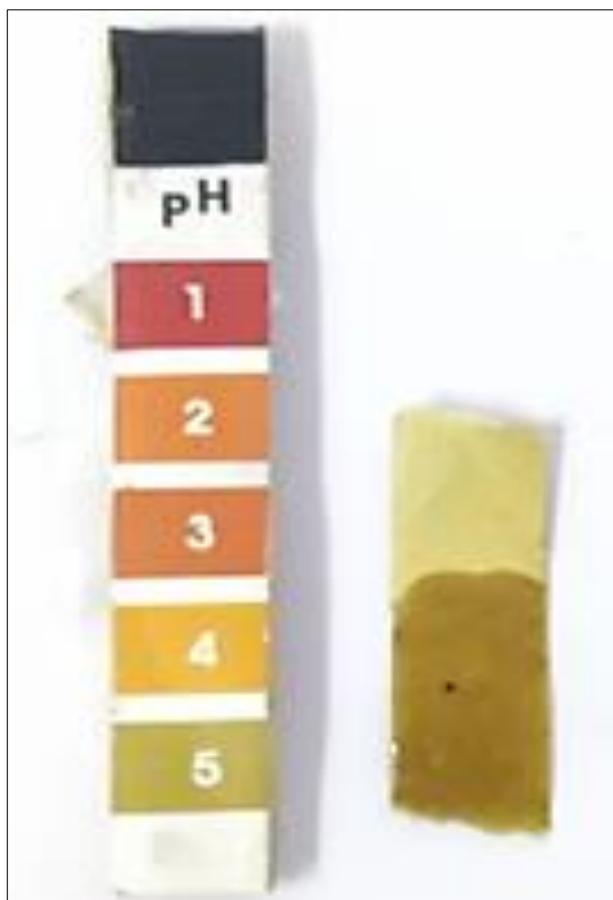
**Physical appearance/visual inspection:** The formulated herbal shampoo was brownish in colour. It has a slight odour.



Fig 4: Colour of the prepared shampoo (physical appearance)

**pH**

The P<sup>H</sup> of formulated shampoo was 6.8, falling within the ideal P<sup>H</sup> range for shampoo which is between 5 and 7.8. The formulated shampoo is acid balanced which is near to the skin P<sup>H</sup>. The P<sup>H</sup> of shampoo is important for enhancing the qualities of hair, stabilizing ecological balance of scalp and minimizing irritation to the eyes.



**Fig 5:** P<sup>H</sup> of the shampoo (The P<sup>H</sup> was found approx. 6.8)

**Percentage of solid content**

If the shampoo has too many solids it will be hard to work into the hair or too hard to wash out. The result of percent of solid content 0.10%.



**Fig 6:** Amount of solid content

**Wetting time**

The wetting ability of a surfactant is dependent on its concentration and is commonly used to test its efficacy. The canvas disk method is a quick, reliable and efficient to evaluate the wetting ability of a shampoo. The wetting time of herbal shampoo was found to be 2.73 secs which is good.



**Fig 7:** Wetting time

**Cleansing action**

The cleansing action was tested on human hairs that had applied oil and not been washed for 7 days. The results of detergency studies showed that the formulation had significant cleansing ability as it was able to remove both dirt and oil from hairs. The silkiness and softness of hairs after hair wash attributes to the conditioning property of the herbal shampoo.

**Foaming ability and foam stability**

Although foam generation has little to do with the cleansing ability of shampoos, it is of importance to the consumer. The final formulation produced stable foams, there was little bit change in foam volume.



**Fig 8:** Height of foam

**Stability Study**

Stability of formulation during the storage period indicated that they are physically and chemically stable. The formulated herbal shampoo is stable at standard room temperature of 25-

30 C. The results indicates that it possesses good stability within two weeks of stability study.



**Fig 9:** Stability study

#### **Skin irritation test:**

The prepared shampoo does not produce any harmful effect on the skin, this is due to the absence of harmful synthetic ingredients. Mostly the synthetic chemicals produce inflammation and irritation to the skin but in this formulation almost all ingredients are obtained naturally.



**Fig 10:** Skin irritation test

#### **Conditioning attributes**

The prepared formulation produced many conditioning benefits such as it improved the lustre of the hair and also provides silkiness and softness to the hair as well as it also helped in increasing mass of the hair.



**Fig 11:** Conditioning action of prepared shampoo

#### **Viscosity**

The viscosity of the shampoo plays an important role in determining the ease of flow on removal from packing and spreading on application to hair, its self-life stability and product consistency in the package. The viscosity of formulated shampoo was found to be 0.67 poise or pascal.sec which was good enough for its applicability.



**Fig 12:** Viscosity testing using Ostwald's Viscometer

#### **Density**

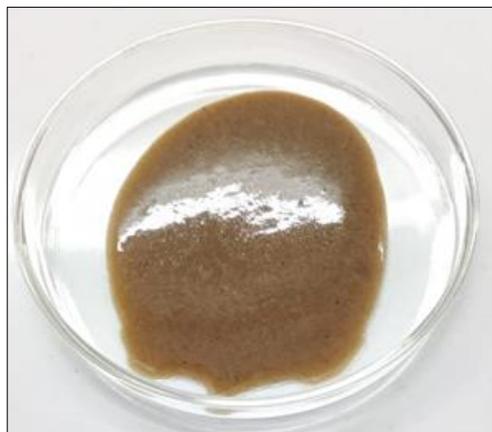
The density of the herbal shampoo was found to be 1.12gm/ml which was good enough for its compactness.



**Fig 13:** Density testing

#### **Microbial examination**

The microbial growth on the formulated shampoo was quite low, so it is safe to keep at room temperature as the shampoo was stable after 24 hours. Therefore, it will not harm the skin or hair of any individual.



**Fig 14:** Microbial growth testing

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### Conclusion

Herbal shampoo using reetha, amla and neem extract is prepared and evaluations were carried out for those following parameters: physical appearance/visual inspection, P<sup>H</sup> determination, determination of solid content percentage, wetting time, cleansing action, foaming ability and foam stability, stability study, skin irritation test, conditioning attributes, viscosity, density, microbial examination. The evaluation parameters data were shown in acceptance range. Further studies are appreciated for comparing this preparation with marketed one and establishing some effective results for hair cleansing action and conditioning effect as well.

### Discussion

The prepared herbal shampoo appeared to be brown and had a slight odour. Its pH was recorded at 6.8 which is in between the ideal range. Parameters that are evaluated for having significant amounts of solid contents in the shampoo. Its wetting time was found to be 4 seconds, its cleansing action was hugely successful, had a good amount of foam formation. The shampoo was kept at room temperature (25-30) and it showed good stability.

Due to inclusion of natural excipients the shampoo doesn't show any sort of skin irritation. To have the consistency, the viscosity was recorded to be 0.67 pois pascal and the density was 1.12g/ml which held out its compactness.

In the span of 24 hours, the shampoo which was being kept at room temperature showed very trace microbial growth which laid it out that the shampoo embarked all the necessary qualities required for the shampoo to pass out for production.

### Authors' Contribution

Ishita Kumari, Ishita Sarkar, Ishika Sanyashi and Rajat Das conceived and planned the experiments. Ishita Sarkar, Sayak Das, Ishika Sanyashi and Ishita Kumari carried out the experiments. All the authors contributed to sample preparation. Ishita Kumari and Ishita Sarkar contributed to the interpretation of the results. Ishita Sarkar and Ishika Sanyashi took the lead in writing the manuscript. All authors provided critical feedback and helped shape the research, analysis and manuscript.

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